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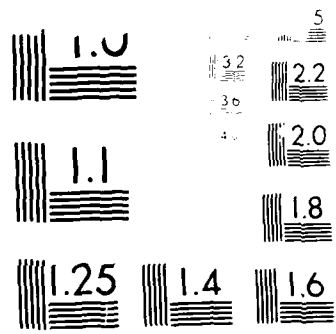
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FINAL REPORT

Development/Applications of FT-IR ATR
and Photoacoustic Dichroism Techniques
for Structural Characterization of Polymer Surfaces

by

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Figure 8. Report Documentation Page, DD Form 1473 (1 of 2)

Objectives

Background

Technical Summary

Toward three-dimensional surface structural analyses by FT-IR ATR dichroism technique, a new ATR attachment was designed and tested. The main feature of this new attachment is a rotatable ATR sample holder which was mounted on a rotating stage. The rotation axis was designed so that it coincided with the entrance plane to the crystal. The angle of incidence could be adjusted by a gear and worm-screw to precise angles (5). This new attachment was tested on well-characterized polymer films (uniaxial, biaxial polypropylene and uniaxial polyethylene terephthalate). From the four possible dichroic ATR spectra, all the necessary optical constants were calculated and used to estimate the surface degree of crystallinity and molecular orientation averaging about a micron in depth. The results on surface crystallinity agreed well with the values from x-ray and DSC studies.

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The ability to alter the angle of incidence accurately with the new ATR apparatus allowed depth profiling from about 1 to 15 microns. The results revealed some subtle changes in orientation and crystallinity as a function of depth on the surface of uniaxially drawn polypropylene (5).

In order to overcome beam divergence and back reflection due to non-integer reflections with face-cut crystals when a wide range of incidence angles is used, as in depth-profiling, a new ATR attachment using a hemispheric crystal had been designed. In this attachment, the sample is held against the crystal surface at a fixed point in space. The goniometer has been designed so as to rotate the crystal horizontally as well as vertically. Under this condition, the resulting beam after a single reflection is always parallel at a precisely known angle of incidence with a constant incident beam energy, regardless of the incident angle. A preliminary study showed promising results (7,8).

In FT-IR PAS dichroism studies, we were able to obtain three dimensional orientation measurement by tilting the sample (45°) in a specially designed PAS cell. The experimental data will be analyzed by extending the theory for the photoacoustic effect on solid surfaces to anisotropic solids.

While we continue to improve and develop new ATR and PAS attachments, our first ATR attachment has been adopted by many laboratories in the world, (e.g. 3M, Rhone-Poulenc, ICI, National Research Laboratory of Canada, University of Washington). Our 3-D ATR attachment has been adopted by Monsanto. They are being used to characterize a variety of polymers and composites such as PEEK and liquid crystalline polymers.

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